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A COMPREHENSIVE PROGRAM FOR AUTOMATING RESIDUAL STRESS MEASUREM--ETC(U)
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A COMPREHENSIVE PROGRAM FOR AUTOMATING RESIDUAL STRESS MEASUREMENTS BY X-RAYS

by

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EVANSTON, ILLINOIS

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ABSTRACT

The software design of a complete computer controlled X-ray residual stress is described. The program will operate using either a position sensitive detector or a diffractometer for data accumulation.

For use with a diffractometer, optimization of the time of data accumulation for a prescribed statistical counting error is presented. The peak location routine, using a multiple pass refinement of the peak position, is detailed.

The software allows operator intervention during operation and specification of either the 'two tilt' or '$\sin^2\psi$' technique. The Marion-Cohen technique is automatically accomplished if oscillations in d vs. $\sin^2\psi$ are evident. Provisions for the receiving slit movement, counting statistical analysis and instrumental error analysis are provided. The operator specifies the total error in stress he will tolerate, and the program then optimizes the counting strategy.

CHAPTER 4*

4.1 INTRODUCTION

Manual measurement of residual stress requires accumulating X-ray counts at a minimum of three 2θ angles over the peak at each ψ inclination of the sample. Long collection times are needed to accumulate the data making the procedure tedious and time consumming. Hardware controlled step scanning may save on operator time, but is of limited value since the peak position and breadth varies with sample, with residual stress and with psi angle. Indeed, the need for operator control has been the chief reason for the proliferation of the 'two tilt' method over the '$\sin^2\psi$' method, although more precision is obtained with the latter.

Instrumentation such as Fastress[76] and the Shimadzu X-Ray Diffraction Stress Analyzer[77] offers limited automation at the expense of precision and versatility. Other automated systems[79] require the collection of many data points in the peak region due to the lack of on line control to locate the peak. None of these instruments offer any type of statistical analysis or optimization of data accumulation.

On line control has been achieved by Kelly and Short[80] and Hayama and Hashimoto[81] by using step scanning techniques. Both use counting statistics to estimate the statistical error, but do not collect data to achieve an operator specified error (i.e., they do not optimize the data collection).

Kelly and Short designed their software package specifically for the 'two tilt' method of X-ray stress analysis and allow only a three

*Ch. 1, 2 and 3 appeared as ONR TR No. 14 on this contract.

point parabolic fit to determine the peak position. Hayama and Hashimoto employed the parallel beam method of X-ray optics and the half-value breadth method to define the diffraction angle.

The system to be described includes the hardware and software design for computer controlled X-ray measurement of residual stress, including sample alignment, optimized data accumulation and on line calculation of the stress and all principal errors. The system is designed for use with either a normal diffractometer with step scanning or with a position sensitive detector (PSD) to collect the data. The operator specifies the error in stress and responds to certain questions on the equipment after which the computer devises the appropriate counting strategy.

4.2 INSTRUMENTATION

4.2.1 Hardware

Control of the diffractometer was executed through a PDP8/E hardware/software system. The basic DEC PDP8/E computer having 16 K, twelve-bit words of memory and dual Dectape transport for mass storage was complimented with interfacing for two detectors, a clock to serve as a time base, and a drive for three SLO-SYN stepping motors. The basic interface arrangement has been described previously[82] and is used for complete automation of the Long-Term X-Ray Facility at Northwestern University. [Because of limited funds, sophisticated items such as absolute angle decoders or addressable axis positions and counters are not incorporated. Therefore, all motor movements and counting procedures are accomplished via software programming. This results in a versatile system that can be easily adapted (by software programming) to control many diverse experiments in the field of X-ray diffraction.]

4.2.2 Software Package

The basis of the software system is a modified version of OMSI-FOCAL tailored specifically to meet the needs of the X-ray facility. OMSI-FOCAL is a Digital Equipment Corporation FORTRAN-like high level language as modified by the State of Oregon Museum of Science and Industry.(83)

User defined functions have been created by the current X-ray group at Northwestern University (including the present author) to allow:

1) Simultaneous movement of three stepping motors.

2) Data accumulation by fixed time, fixed counts, or fixed monitor counts.

3) Accumulation of data from two detectors simultaneously.

4) Two hundred ninety-six directly addressable variables and 992 indirectly addressable arrayed variables.

5) Accumulator switching to enable control of a teletype, digital voltmeter, thermac controller, and a multichannel analyzer interface. This function allows a change in the operating status of the teletype (on-off) or a Thermac controller (from a low set point to a high set point), monitoring the readout of a digital voltmeter, and transfer of data from a multichannel analyzer to the computer.

This version of Focal, designated by the X-ray group as X-RAY FOCAL, was designed to alleviate any additional machine language programming in automating X-ray diffraction experiments. The computer program, STRESS, to automate the residual stress measurement is described in Sec. 4.3.

4.3 PROGRAM FEATURES

4.3.1 Introduction

The following features are implemented in the automated residual stress package called STRESS.

a. Automatic sample alignment.

b. Use of either 'two tilt', '$\sin^2\psi$' or 'Marion-Cohen' methods of residual stress analysis (see Sec. 4.3.4).

c. Use of a step-scanning diffractometer or a position sensitive detector system for data accumulation.

d. Parafocusing or stationary slit 'beam optics' (see Sec. 4.3.5).

e. A least-squares parabola to determine the peak location (see Sec. 4.3.6).

f. On-line peak location with operator specified accuracy (see Sec. 4.3.7 and 4.3.8).

g. Specification of experimental elastic constants or single crystal elastic constants (see Sec. 4.3.9).

h. Instrumental error calculations (see Sec. 4.3.10).

i. Plot of data for '$\sin^2\psi$' and 'Marion-Cohen' methods.

j. Oscillation of sample (see Sec. 4.3.2).

A master flowchart describing the overall sequence of subprograms is given in Fig. 4.1. A thorough description of each feature of the program follows. The master dialog sets operating parameters for the entire residual stress package and is explained in Sec. 4.3.2. The program may branch to a sample alignment program or the initial dialogue

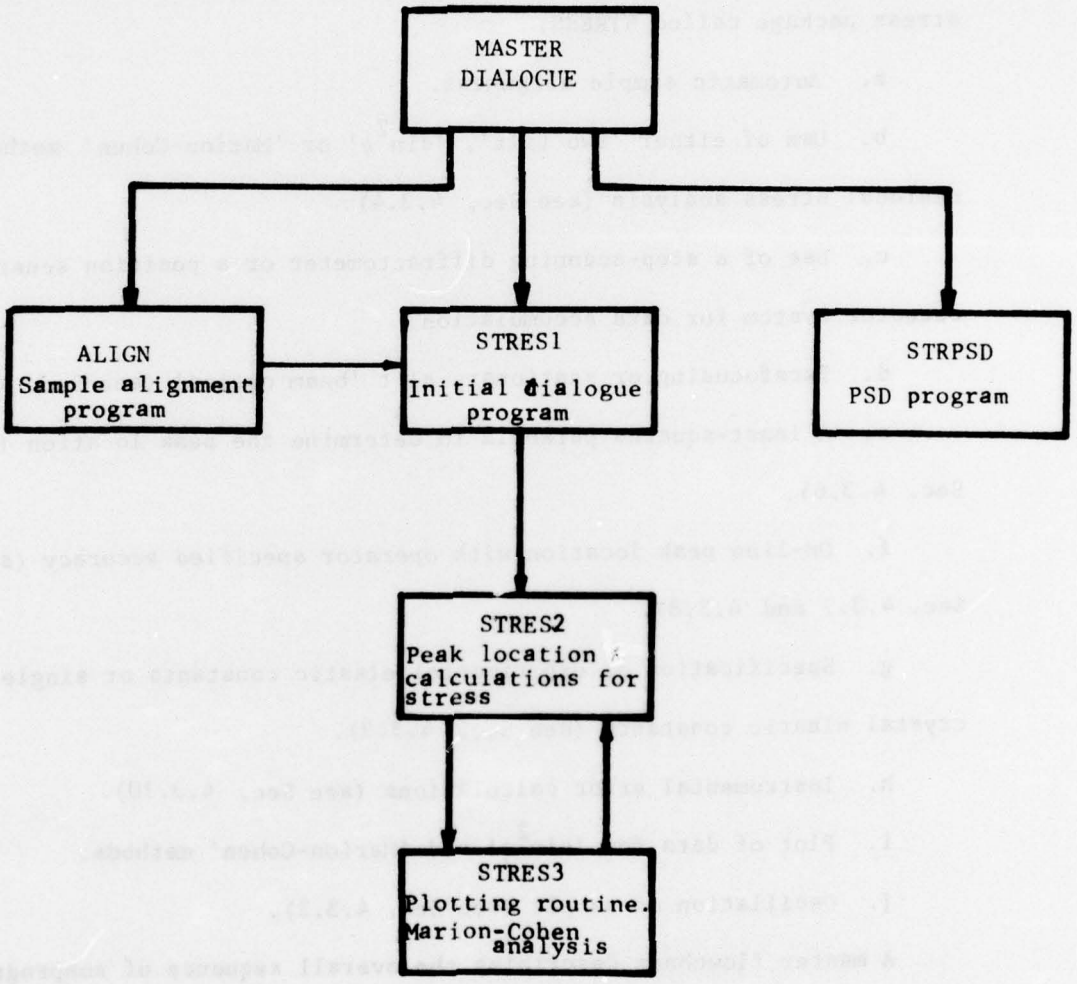

FIGURE 4.1 Flowchart for program calling sequence in the residual stress package.

for the residual stress program using either a step scanning diffractometer or a position sensitive detector. It should be pointed out that, as seen in Fig. 4.1, if the stress program using the PSD, STRPSD, is being used, the sample alignment program cannot be implemented. The PSD needs to be calibrated to change linear position along the detector into $^{\circ}2\theta$. This would not be practical in determining peak positions for multiple peaks because an absolute 2θ along the PSD would not be known once the PSD was moved. Thus, the PSD is only practical for repetitive measurements on similar samples or for use on a dedicated residual stress device.

Sample input and output are provided for each subprogram with flowcharts detailing specific sections. Line numbers on the input-output figures are referred to in the text for convenience to the reader.

4.3.2 Master Dialogue

The master dialogue sets parameters which are used in the five subprograms of the residual stress package and chains to the appropriate subprogram when complete. A sample input is given in Fig. 4.2.

The master program chains to the position sensitive detector subprogram if a positive response is given to line 2 of Fig. 4.2. The sample alignment program is run if a positive response is given to line 9 in Fig. 4.2.

Two options affecting the stepping motor pulse rate are necessary for the package to be used on all diffractometers in the X-ray facility

```
.R STRESS

X-RAY LAB FOCAL V2   12/19/76


                    AUTOMATED RESIDUAL STRESS PROGRAM
                 (COPYRIGHT  NORTHWESTERN UNIVERSITY, 1976)

     TURN OFF TTY
1    SHORT DIALOG ? <Y/N> N

2    USING POSITION SENSITIVE DETECTOR ? <Y/N> N
3    EXPT. IN ROOM B-893 ? <Y/N> Y
4    EXPT. USING PICKER DIFFRACTOMETER ? <Y/N> Y
5    USING SOLID STATE DETECTOR ? <Y/N> Y   DELAY TIME IS (SEC) ?
6    DEADTIME (SEC) 1.5E-6
7    OSCILLATE FEATURE ? <Y/N> Y
8       ROCKING WIDTH (DEGREES 2THETA) 5

9    DO YOU WANT TO ALIGN SAMPLE ? <Y/N> Y



     -CHECK LIST-
     1.  BE SURE MOTOR DIRECTION SWITCHES ON COMPUTER ARE
         PROPERLY SET AND VOLTAGE LEVEL IS SET TO 16 V.
     2.  TTY 'ON/OFF' CABLE PLUGGED IN
     3.  LIMIT SWITCHES SET (BE SURE DETECTOR CLEARS
         X-RAY TUBE AT HIGH 2THETA)
     4.  SAMPLE ALIGNMENT CHECK LIST
            A. IF AUTOMATED REPOSITIONING IS USED BE SURE
               MOTOR IS CONNECTED TO THE PICKER SAMPLE HOLDER.
            B. USE ALIGNMENT TOOL TO ROUGHLY SET POSITION.
               ---BE SURE TO CLOSE SHUTTER BEFORE USING TOOL--
            C. DO YOU NEED COMPUTER TO ROUGHLY ALIGN <Y/N> ? Y
               GIVE 'INCREMENT' TO BE MOVED
               2THETA     PSI    SAMPLE
              DEGREES  DEGREES  INCHES
               -20        0      .05          REPEAT <Y/N> ? N

     TYPE ALT MODE TO CONTINUE
```

FIGURE 4.2 Sample output from the master dialogue.

at Northwestern. By responding to lines 3 and 4 in Fig. 4.2 with alphanumeric responses of a 'Y' for yes or a 'N' for no the program sets the proper pulse speed to the motors.

Line 5 of Fig. 4.2 determines if a solid state detector is to be used. Because of the vibration due to the movement of liquid N_2, a delay between movement of the detector and the counting of photons is made to prevent the detection of noise generated in the detector. The delay time is input by the user in line 5, Fig. 4.2. The deadtime of the counting system is input in line 6.

Specimen oscillation may be used to increase the number of crystals involved in diffraction for coarse grained materials. This method is based on the idea that the stress measured by X-ray diffraction is the value averaged over the area illuminated. Small oscillations in the ψ axis will not greatly affect the result since the average value of $\sin^2\psi$ will not change at each peak. To initiate this feature, the user responds with a 'Y' to line 7 of Fig. 4.2 and then inputs the rocking width of the oscillation. The axis of oscillation is the ψ axis.

An option is given in line 1 of Fig. 4.2 to skip all these questions. This default scheme assumes the use of the Picker diffractometer with a scintillation detector, and sets the deadtime to 1.5 μsec. The program chains directly to the initial dialogue for the residual stress program using a diffractometer or to the sample alignment program if requested.

4.3.3 Sample Alignment

The displacement of the sample from the true center of the diffractometer can be estimated from the slope of the lattice parameter vs. Nelson-Riley

factor. A subprogram, ALIGN, automatically locates the Bragg angle of any peak by defining the peak position from the apex of a parabola fit to the top 15 pct of the diffraction profile. After determining three or more peaks specified by the operator, the lattice parameter and Nelson-Riley factor are determined. The extrapolated lattice parameter is determined from a linear least-squares fit to the lattice parameter vs. Nelson-Riley factor. The sample displacement is determined from the slope of the least squares fit after which the operator may manually or automatically reposition the sample and repeat the alignment or chain to the initial dialogue for the diffractometer version of the automated residual stress program.

Figure 4.3 reproduces the input and output of the alignment program. Only three data points are used in the parabolic fit. The operator needs only specify the current angular settings, the initial 2θ position from where to begin the first step scan to find the rough peak location and the 2θ step increments for the first and second step scans (designated DELTA 1 and DELTA 2, respectively) to refine the peak position. This peak location scheme is identical to that used in the residual stress program, a detailed description of the procedure being given in Sec. 4.3.7. The only change is that the operator has the option in the alignment program to specify the accuracy of each peak location (in °2θ). If he does not, a fixed count of 10,000 is obtained for each data point.

The peak location and the error on the peak location (one standard deviation) are output. For convenience the peak intensity and the step

```
                    -SAMPLE ALIGNMENT PROGRAM--

        CURRENT
      2THETA    PSI      2THETA MAX     2THETA MIN  ZERO 2THETA
      134.60    0.0         162.5          64.0         -.005

 SPECIFY ACCURACY <Y/N>     N
 WEAK PEAK FEATURE ? <Y/N> Y
 GIVE PRESET COUNTS FOR PEAK SCANS
 1ST PRELIMINARY SCAN 500
 2ND PRELIMINARY SCAN 1000
 FINAL SCAN           4000
 NUMBER OF PEAKS         3

 ID   INITIAL 2THETA   DELTA 1   DELTA 2
   1       68.4           .1        .02
   2       105.5          .15       .02
   3       155            .20       .04          CHANGE ANY DATA <Y/N> N

CHECK LIMIT SWITCHES - OPEN SHUTTER
TYPE ALT. MODE TO CONTINUE

  ID   PEAK LOCATION     ERROR      PEAK INT      DELTA

   1        68.815       0.001323   2514.46       0.075
   2       106.130       0.004477    327.26       0.210
   3       156.273       0.006994    996.37       0.360

FINISHED - TURN ON TTY - TYPE ALT. MODE TO CONTINUE

   2THETA     H K L   WAVELENGTH       D          LP        N-P FACTOR
  68.815      1 1 0     2.2909      2.027080   2.866724    1.169052
 106.130      2 0 0     2.2909      1.433032   2.866063    0.420911
 156.273      2 1 1     2.2909      1.170451   2.867007    0.237088

CHANGE ANY DATA <Y/N> N

LATTICE PARAMETER =          2.866649        (ST. DEV. =   0.000600)
SLOPE OF REGRESSION LINE = - 0.000094
CORRELATION COEFFICIENT =  - 0.987360

GONIOMETER RADIUS (INCHES) ? 5.73

SAMPLE DISPLACED TOO FAR FORWARD BY  0.0002 INCHES
MANUAL OR AUTOMATED SAMPLE REPOSITIONING ?
MANUAL ? <Y/N> N
AUTOMATED REPOSITIONING COMPLETE

REPEAT MEASUREMENT <Y/N> ? N

RUN STRES1.FC ? <Y/N> Y


TURN OFF TTY
```

FIGURE 4.3 Sample input/output for the alignment program.

increment between the final data points, DELTA, are printed. If the operator desires to repeat the alignment; e.g., if he repositions the sample and wants to check it, this information enables him to judge more accurately the precision of peak location and the two step increments.

The extrapolated lattice parameter, slope and correlation coefficient from the lattice parameter vs. Nelson-Riley plot are printed. The sample displacement is typed after the operator gives the goniometer radius. The alignment program may be repeated or the initial dialog program for the diffractometer version for residual stress determination may be run.

4.3.4 Method of Residual Stress Analysis

The 'two tilt', '$\sin^2\psi$' or 'Marion-Cohen' methods of residual stress analysis are available by responding with a 'Y' for 'yes' to the appropriate question (lines 1-3 in Fig. 4.4*). The specifics of each method are detailed below. This applies to both the diffractometer and position sensitive detector version.

(A) 'Two Tilt' Method

If the operator chooses this method only two tilts of the specimen are employed to determine the residual stress. The ψ angles are requested for each tilt (line 19-20, Fig. 4.5). The stress is determined using either an exact formalization or an approximate equation relating the stress directly to the peak shift through a stress constant, K. The choice is made when the operator inputs the elastic

*Because of the many options, two input dialogs are given in Fig. 4.4 and Fig. 4.5. Line numbers and the figure number are given wherever a reference to each figure is given. Typical output is given in Fig. 4.6 for the two tilt and Fig. 4.7 for the $\sin^2\psi$ and Marion-Cohen method.

```
                    -DIFFRACTOMETER VERSION OF STRESS-

     -METHOD OF RESIDUAL STRESS ANALYSIS-
1    TWO TILT METHOD      <Y/N>  N
2    SIN**2 PSI METHOD    <Y/N>  Y
3    MARION-COHEN METHOD  <Y/N>  Y
4    REC. SLIT MOVEMENT   <Y/N>  N

     -CURRENT SETTINGS-
5    2THETA                      156.675
6    PSI                         0.0
7    TRUE ZERO 2THETA            -.005

     -PRELMINARY SCAN DATA-
8    APPROX PEAK 2THETA          156
9    INITIAL 2THETA              154
10   1ST INCREMENT               .20
11   2ND INCREMENT               .07
12   BACKGROUND FEATURE <Y/N> ?  N
13   WEAK PEAK <Y/N> ?           N

     -FINAL SCAN DATA-
14   NUMBER OF DATA POINTS       5
15   WAVELENGTH                  2.2909
16   SCATTERING FACTOR CORR. ? <Y/N> N

     NUMBER OF PSI TILTS         4
17   PSI(  1 )  0
18   PSI(  2 )  26.57
19   PSI(  3 )  39.23
20   PSI(  4 )  50.77

21   MARION-COHEN METHOD
         SPECIFY INCREMENT (DEGREES 2THETA) BELOW
         PEAK FOR BACKGROUND DETERMINATION :   6
         LIMIT THE DIFFRACTED BEAM HEIGHT AT THE DETECTOR
         TO BE .3 INCHES FOR TEXTURE ANALYSIS

     -- ELASTIC CONSTANTS DATA --
22   UNITS OF PSI (TYPE 1) OR MPA (TYPE 2) :2
23   EXPT. ELASTIC CONSTANTS ? <Y/N> N

     SINGLE CRYSTAL ELASTIC CONSTANTS IN MM**2/KG
24   S11 .00007845
25   S12 -.00002746
26   S44 .00008543

     H K L              WEIGHT                  STRESS                  AVERAGE
27   2 1 1   S1    -0.1085869419E-04    -0.1166125000E-04    -0.1125997209E-04
             S2/2   0.5612628255E-04     0.5851374999E-04     0.5730991627E-04
28   (1+NU)/E =  2.5844372452E-05 MPA


     -- TOTAL ERROR --
29   ERROR IN  MPA ? <Y/N> Y     12

     -- INSTRUMENTAL ERROR --    <Y/N> ? Y
30   DIVERGENCE SLIT (DEGREES)   1
31   SAMPLE DISPLACEMENT         .005
32   PSI AXIS MISSETTING         0
33   GONIOMETER RADIUS           5.73

34   INSTRUMENTAL ERROR APPROX    3.61 MPA      (   523 PSI )

35   COUNTING ERROR IN 2THETA IS   0.0293

36   HOW MANY REPEATED MEASUREMENTS    1

37   CHECK TOTAL TIME <Y/N>            N

     CHECK LIMIT SWITCHES AND SHUTTER
     HIT ALT MODE TO CONTINUE
```


FIGURE 4.4 Sample input for diffractometer version of STRESS. Data is input for a typical measurement using the $\sin^2\psi$ technique. Copyright Northwestern University, 1976.

```
                    -DIFFRACTOMETER VERSION OF STRESS-

     -METHOD OF RESIDUAL STRESS ANALYSIS-
1    TWO TILT METHOD      <Y/N>  Y
2    REC. SLIT MOVEMENT   <Y/N>  N

     -CURRENT SETTINGS-
3    2THETA                      158.17
4    PSI                         50.77
5    TRUE ZERO 2THETA            0.0

     -PRELMINARY SCAN DATA-
6    APPROX PEAK 2THETA          156
7    INITIAL 2THETA              154.5
8    1ST INCREMENT               .25
9    2ND INCREMENT               .10
10   BACKGROUND FEATURE <Y/N> ? Y          BKGD ANGLE : 148.0
11   WEAK PEAK <Y/N> ?           Y
12   GIVE COUNTS FOR 1ST 2 SCANS  CT(1)=400   CT(2)=1000

     -FINAL SCAN DATA-
13   NUMBER OF DATA POINTS       3
14   WAVELENGTH                  2.2909
15   SCATTERING FACTOR CORR. ? <Y/N> Y
16   GIVE SCAT. FACT. AT TWO POSITIONS IN SIN(THETA)/LA NEAR PEAK

              SIN(THETA)/LA  SCATTERING FACTOR
17                0.50           11.47
18                0.40           13.84

     -PSI TILTS-
19   PSI(  1 )  0
20   PSI(  2 )  45

     -- ELASTIC CONSTANTS DATA --
21   UNITS OF PSI (TYPE 1) OR MPA (TYPE 2) :1
22   SPECIFY STRESS CONSTANT ? <Y/N> Y
     K = PI/180 * E/(1+V) * 1/SIN^2(PSI) * COT(THETA)
23   K ( PSI/'2THETA)            86200

     -- TOTAL ERROR --
24   ERROR IN  PSI ? <Y/N> N
25   ERROR (2THETA)              .015

26   -- INSTRUMENTAL ERROR --   <Y/N> ? N

27   HOW MANY REPEATED MEASUREMENTS    1

28   CHECK TOTAL TIME <Y/N>            N

     CHECK LIMIT SWITCHES AND SHUTTER
     HIT ALT MODE TO CONTINUE
```

FIGURE 4.5 **Sample input for diffractometer version of STRESS. Data input for a typical measurement using the two-tilt technique.**

```
PSI=    0.00                          BKGD INT =        118 CPS
  ANGLE          CPS      CORR FACTOR      CORR CPS       TIME
  155.570       324.97      3.62425          89.665      17.477
  156.195       571.67      3.67130         155.713       9.935
  156.820       438.33      3.71876         117.871      12.957

PEAK LOCATION   156.280
ST. DEV.        0.00766


PSI=   45.00                          BKGD INT =         84 CPS
  ANGLE          CPS      CORR FACTOR      CORR CPS       TIME
  155.505       280.41      2.83370          98.957      13.896
  156.055       385.91      2.88445         133.791      10.292
  156.605       324.30      2.93583         110.462      12.207

PEAK LOCATION   156.189
ST. DEV.        0.01323


STRESS                      101.07 MPA   (     14658 PSI )
STATISTICAL ERROR     +/-     9.06 MPA   ( +/-  1314 PSI )
```

FIGURE 4.6 Sample output for STRESS package using the two-tilt technique. Background feature was used and scattering facter correction made to the raw data.

```
PSI=    0.00
  ANGLE          CPS      COPR FACTOR     COPR CPS      TIME
  155.860       742.81      1.91655        387.576      12.269
  156.055       861.23      1.91781        448.964      12.412
  156.250       905.10      1.91906        471.638       9.905
  156.445       831.40      1.92230        432.353      12.783
  156.640       754.82      1.92154        392.819      11.877

PEAK LOCATION   156.247
ST. DEV.        0.00691


PSI=   26.57
  ANGLE          CPS      COPR FACTOR     COPR CPS      TIME
  155.645       612.30      1.70877        358.393      14.485
  155.875       900.40      1.71181        467.574      11.281
  156.105       882.07      1.71515        514.282      12.755
  156.335       834.44      1.71848        485.567      12.629
  156.565       750.23      1.72180        435.725      11.822

PEAK LOCATION   156.176
ST. DEV.        0.00693


PSI=   39.23
  ANGLE          CPS      COPR FACTOR     COPR CPS      TIME
  155.550       491.97      1.57586        312.192      14.424
  155.835       657.06      1.58146        415.478      10.830
  156.120       715.93      1.58705        451.108       9.912
  156.405       633.37      1.59262        397.688      11.204
  156.690       571.31      1.59819        357.472      12.421

PEAK LOCATION   156.159
ST. DEV.        0.00748


PSI=   50.77
  ANGLE          CPS      COPR FACTOR     COPR CPS      TIME
  155.585       428.86      1.40739        304.723      17.677
  155.805       512.16      1.41315        362.424      14.822
  156.025       533.16      1.41892        375.751      14.219
  156.245       500.93      1.42467        351.607      15.134
  156.465       470.17      1.43043        328.691      16.124

PEAK LOCATION   156.054
ST. DEV.        0.00979


 SIN^2 PSI        D(I)          ERROR D(I)
    0.000       1.170506       0.0000212
    0.200       1.170660       0.0000211
    0.400       1.170697       0.0000228
    0.600       1.170923       0.0000300

SLOPE                       0.00064424
INTERCEPT                   1.17050314
COPR COEFF                  0.94479364
STAN DEV OF SLOPE           0.00012649

STRESS                        94.18 MPA    (     13659 PSI )
STATISTICAL ERROR     +/-      8.35 MPA    (  +/-  1211 PSI )
INSTRUMENTAL ERROR             3.58 MPA    (     519 PSI )

                    TEXTURE ANALYSIS


  PSI        IPEAK        IBKGD     IP/IB
             CORRECTED
    0.000    900.790     198.883    1.022
   26.570    911.334     175.213    0.901
   39.230    589.130     156.765    0.655
   52.770    565.434     137.711    0.628


B1=C0=(1+V)/E=STRESS     B1=   0.000857    (SD OF B1=   0.274714)
B2=CMAX-CB               B2=   0.289109    (SD OF B2=   0.226637)
B0=   1.16320


STRESS                 114 MPA          (     16511 PSI)
CMAX              1.172296
CB                1.163196
```

FIGURE 4.7 Sample output for STRESS package using the $\sin^2\psi$ method with Marion-Cohen texture analysis.

constants (lines 21-23, Fig. 4.5).

The statistical counting error in the stress for the two tilt method is calculated

depending on whether an exact or approximate formalization is used.

(B) $\sin^2\psi$ Method

Up to eight ψ tilts are allowed when using the $\sin^2\psi$ method, as shown in lines17-20, Fig. 4.4. The stress is calculated and the statistical counting error in stress determined. A linear least squares method given by Ezekiel and Fox[85] is utilized to calculate the slope of d_ψ vs. $\sin^2\psi$. The intercept, correlation coefficient and standard deviation of the slope are calculated and printed. A plot of d_ψ vs. $\sin^2\psi$ is given if requested.

(C) Marion-Cohen Method

The use of the Marion-Cohen method to enable the determination of the macroscopic residual stress in the presence of oscillations in d_ψ vs. $\sin^2\psi$ has been presented. All features of the $\sin^2\psi$ method are incorporated in this technique. After the normal $\sin^2\psi$ analysis, the correlation coefficient[86] is tested to judge if the fit of d_ψ vs. $\sin^2\psi$ is linear within a 90 pct confidence level or if oscillations may be present. If the latter is true, the Marion-Cohen analysis is implemented.

The required distribution or texture function is determined using the peak intensity at each ψ tilt. Both the maximum

and background intensity are obtained with the receiving slit positioned on the goniometer circle. This is to insure that the same size arc on the Debye cone is recorded for each tilt which allows for normalization of the distribution function.[78] The background is subtracted from the maximum intensity and the net value corrected for absorption* as described by Marion.[30] These values are normalized with the maximum value as unity. A multiple least-squares fit is performed and $(d_{max}-d_B)$, d_B, and the stress are calculated. A typical output is given in Fig. 4.7.

4.3.5 Focusing Technique

The operator is queried whether or not he wishes to have the receiving slit moved in line 4, Fig. 4.4. This question effectively determines if parafocusing geometry (movement of the receiving slit) or non-focusing geometry is implemented. An approximate peak position given by the operator in the initial dialog, line 6 of Fig. 4.5, is used in the calculation of the distance of movement. Instrumental errors are calculated using the appropriate sample to receiving slit distance determined for each technique.

4.3.6 Number of Data Points in Peak Location

Independent of the method of X-ray stress analysis, the operator specifies the number of observation positions for the parabolic fit in line 14 of Fig. 4.4. At least three data points must be used to fit a parabola to the profile. A least-squares routine is used to locate the apex of the parabola and requires only an odd number of data points.

*The absorption correction is necessary because of the change in the area of the specimen irradiated in going from one ψ angle to another and the relative distances travelled by the incident and diffracted rays through the specimen.

4.3.7 On-Line Peak Location

Automated location of the peak of a diffraction profile requires first determining the region of fit of a smooth curve to the profile, obtaining the data at the proper angular positions to the desired precision and processing the data. A flowchart covering the peak location is given in Fig. 4.8.

The region of fit for a parabola is chosen using the 'top 15 pct rule'.[26] An option is supplied (line 10, Fig. 4.5) allowing the background to be subtracted prior to determining the region.

The angular position at which the background intensity is to be obtained must be input. A constant value for the background is used because the correction is only made in determining the region of parabolic fit and is not crucial.

An approximate position for the maximum in intensity is found utilizing a step scan initiated from an angular position (input in line 6, Fig. 4.5) below the peak. A large step increment, specified by the operator in line 7 of Fig. 4.5, may be used to speed up this first scan. A fixed count of 1000 is used unless the operator desires to decrease the preset counts if a weak peak is being examined (line 11, Fig. 4.5). The maximum intensity is stored with the scan continuing until a count rate of less than 90 pct of the maximum is found. This allows a 99 pct (three sigma) confidence in the statistical count rate (if 1000 counts are used) to be sure the maximum has been passed. With the maximum count rate and its angular position stored, two step scans down each side of the peak in smaller step increments are initiated. The increment for this second step scan to further refine the peak is

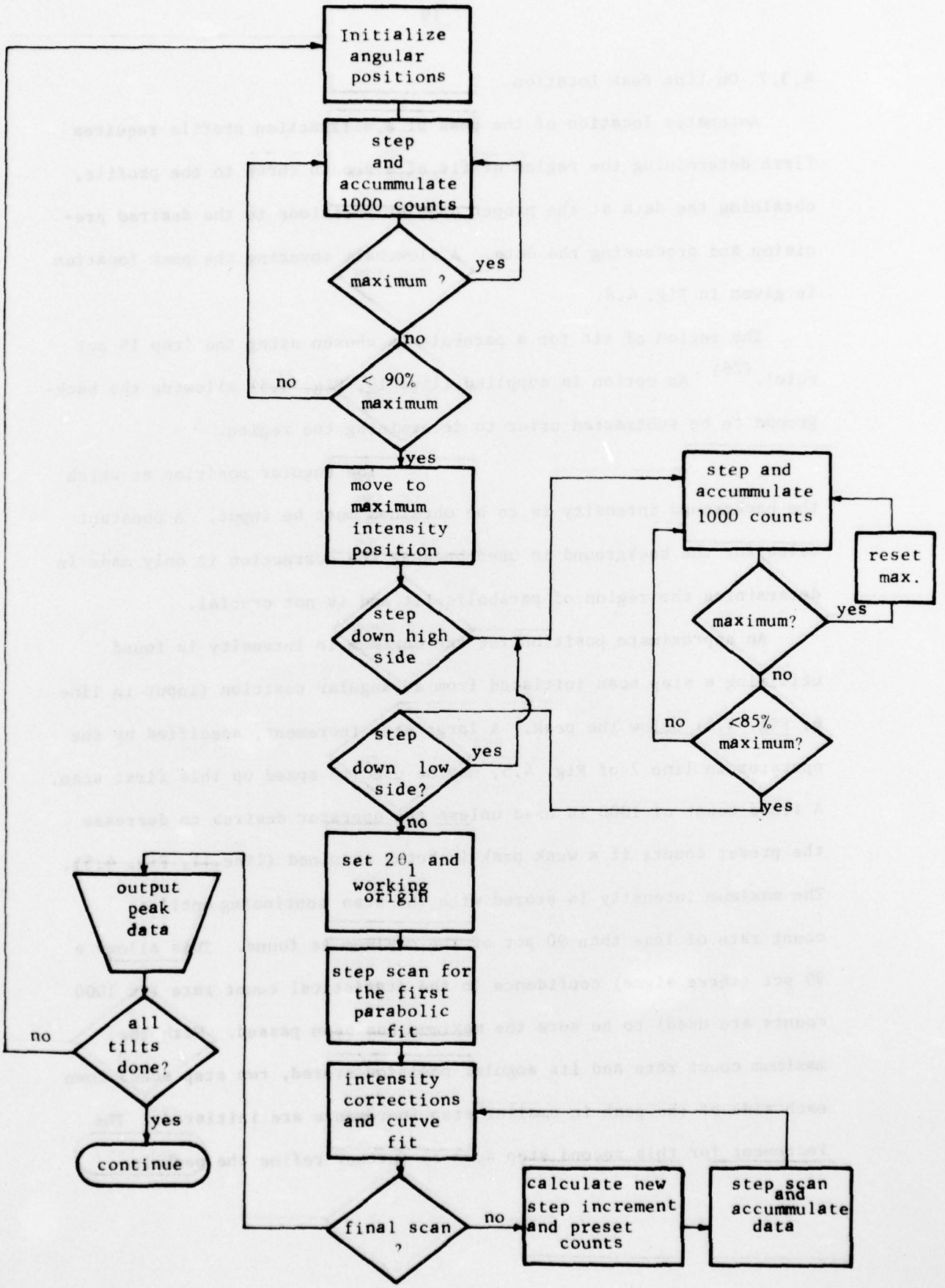

FIGURE 4.8 Flowchart for peak location

specified in line 9, Fig. 4.5. The maximum count rate is adjusted if a greater intensity than heretofore assigned to the maximum is obtained at any position during the second step scan using the smaller step increments. The step scans continue until angular positions, $2\theta_1$ and $2\theta_3$, corresponding to 85 pct of the maximum intensity (with background subtracted if desired) are located. These two positions, $2\theta_1$ and $2\theta_3$, fix the beginning and end of the region of the parabolic fit.

(A) Data Accumulation

Using the two end points, $2\theta_1$ and $2\theta_3$, and the working origin as half way between, a preliminary three point parabolic fit, using fixed counts of 5000, is made. All angular intervals are rounded to the nearest .005°2θ when they are calculated because the 2θ SLO-SYN motor can only be positioned to this accuracy. The data is corrected for Lorentz-polarization, absorption and scattering factors and a parabola fit to the data is obtained. Based on this parabola the angular positions having 85 pct of the peak intensity are determined. The step increment is determined using the desired number of data points (see Sec. 4.3.6). The scan is repeated once more using the desired number of observation points to obtain data on which to estimate the necessary preset counts to achieve the desired accuracy (see Sec. 4.3.8). The final data scan is made using the same angular positions as the previous scan but for the calculated preset counts.

Based on numerous preliminary tests, this method of five refining step scans was judged to be the fastest and most reproducible method of determining the peak position, especially for broad peaks. In summary, the five scans are:

1) Find approximate peak using large step increments. This normally takes about 30 seconds, depending on the intensity.

2) Find region of fit using small step increments. For a sharp peak this takes approximately 30 seconds and 60 seconds for a broad peak.

3) Refine peak location using parabolic fit to three angular positions in region of fit (~ 60 seconds).

4) Obtain data rapidly at final angular positions to determine necessary preset counts (~ 30 seconds).

5) Obtain final data.

(B) Processing of Data

The raw data is corrected for the intensity correction factors for the Lorentz-polarization absorption factors, respectively. Correction for the atomic scattering factor is implemented when desired by the operator. The operator must specify two values of the scattering factor at their respective value of $\sin\theta/\lambda$ as indicated in lines 17-18, Fig. 4.5. A straight line is fit to this data and the slope used to calculate the appropriate scattering factor at each angular position. The data is <u>divided</u> by the Lorentz-polarization, absorption and scattering factor squared. The temperature factor is not included because it is so insensitive to 2θ but this would be included if the operator inputs $f e^{-M}$ when asked for f.

Peak location is determined with the counting statistical error in the peak calculated for fixed count data accumulation. A report of the peak angular position, intensity at each observation point, the appropriate correction factor, the time of data accumulation at each point and the statistical counting error is typed.

This procedure of peak location and output is continued until all ψ tilts are complete. Finally, the residual stress, counting error and instrumental error* are calculated and a report typed. A typical output is given in Fig. 4.6 for the two tilt method and Fig. 4.7 for the $\sin^2\psi$ and Marion-Cohen methods.

4.3.8 Optimization of Data Collection for a Specified Accuracy

In order to determine the appropriate counting time, the program must be told the desired accuracy in the peak position. The program allows the error to be input in the initial dialog (line 29, Fig. 4.4) in terms of $^\circ 2\theta$ for the peak location or in terms of stress from which the appropriate error in the location of each peak is determined.

In the first case, direct input of the accuracy for the peak location, the operator specifies in $^\circ 2\theta$ the precision to which each peak should be located. One value is input and each peak located to that accuracy using only the counting statistics. This is shown in lines 24-25, Fig. 4.5.

If the total error is given in terms of stress, as in line 29, Fig. 4.4, the computer must back calculate the error in peak location as follows:

a) Estimate the instrumental error. This is done using the approximate peak location as specified by the operator and other data given by the operator (see Sec. 4.3.10 for discussion of instrumental error input).

b) The error, E, in stress due to counting statistics alone is then calculated from:

$$E_{(\text{counting statistics})} = \left[E^2(\text{TOTAL}) - E^2(\text{INSTRUMENTAL ERROR})\right]^{\frac{1}{2}} \qquad (4.1)$$

*The exact instrumental error and the correct sign is printed. This may be subtracted from the measured stress (if the input parameters are correct) or may treated simply as an additional error. Eq. 4.1 may be used to obtain a total error if the latter treatment is preferred.

c) The error in peak location is then back calculated from the counting statistical error (which is in units of stress) using equations pertaining to the choice of the measurement.

The calculations are made to give the same statistical error at each ψ tilt. The computer types out the error in the peak location to be sure it is acceptable to the operator. This error represents one standard deviation confidence limits and all peaks are determined to this precision.

The actual determination of the preset counts takes place in the fourth step scan (see Sec. 4.3.7). From the third step scan, the final angular positions are determined. In the fourth step scan, data is taken rapidly at these angular positions. Using this data the summations necessary to derive the error in peak location for fixed counts can be calculated. Setting the specified counting error equal to $\sigma^2(2\theta p)$ enables the minimum preset counts to obtain the desired accuracy to be calculated. The final data is taken for the necessary fixed counts.

This process optimizes the time of data collection for a specified accuracy. The calculation time is minimal and, because all but the fourth step scan are necessary to determine the position of the observation points, very little extra time is taken to optimize the final data accumulation. The fourth step scan, usually taking approximately 30 sec., is necessary to obtain an accurate prediction of the summation term.

Preliminary tests showed that if the data obtained in the third step scan is used to back calculate the fixed counts the estimate of the preset counts is not accurate because the angular positions of the data points are different between the third and final step scans.

If the operator requests an estimate of the total time (line 18, Fig. 4.5) the program will print the total length of time of the measurement based on the time necessary to obtain the first peak. At this point, the operator may change the counting error if the time is not acceptable from which a new total time of analysis, based on the current data, is calculated by the computer and printed out. This sequence is repeated until the operator accepts the total time and operation continues.

4.3.9 Specification of the X-Ray Elastic Constants

The operator may specify the elastic constants in one of three ways and may use units of either MPa or PSI as shown in line 21 of Fig. 4.5.

If the approximate formula for the two tilt method is to be used the stress constant, K, must be input. The stress constant depends on the ψ inclination and it is up to the operator to use the proper tilt.

The best method to input the elastic constants is to use measured values from the literature. These must be input as $(1+\nu)/E$ and may be determined from mechanically measured bulk elastic constants or preferrably from values measured by X-ray diffraction. A good list is given in ref. 90.

The single crystal elastic constants may be used to calculate the elastic constants of a polycrystalline material [11] (see lines 24-27, Fig. 4.4). The theoretical methods vary according to the fundamental boundary conditions used to account for the influence of elastic anisotrophy and grain interactions. A satisfying solution applicable to the

X-ray elastic constants has not yet been achieved.[87] Modified approximations of Voight[74] and Ruess[75] for the calculation of elastic constants for quasi-isotropic polycrystals are employed. The equations for the Voight model are:

$$-\nu/E = (S_1)^{\text{VOIGHT}}_{\text{X-RAY}} = \frac{S_{11}(2S_{11} + 2S_{12} - S_{44}) + S_{12}(3S_{44} - 4S_{12})}{2S_{44} + 6(S_{11} - S_{12})} \tag{4.2}$$

and

$$\frac{1+\nu}{E} = (\tfrac{1}{2}S_2)^{\text{VOIGHT}}_{\text{X-RAY}} = \frac{5\,(S_{11} - S_{12})\,S_{44}}{2S_{44} + 6(S_{11} - S_{12})} \tag{4.3}$$

where S_{ij} are the elastic-compliance constants for the corresponding single crystals. The analysis by Ruess yields:

$$-\nu/E = (S_1)^{\text{RUESS}}_{\text{X-RAY}} = S_{12} + \Gamma\,(S_{11} - S_{12} - \tfrac{1}{2}S_{44}) \tag{4.4}$$

and

$$\frac{1+\nu}{E} = (\tfrac{1}{2}S_2)^{\text{RUESS}}_{\text{X-RAY}} = S_{11} - S_{12} - 3\Gamma\,(S_{11} - S_{12} - \tfrac{1}{2}S_{44}) \tag{4.5}$$

where

$$\Gamma = \frac{h^2k^2 + k^2\ell^2 + \ell^2h^2}{(h^2 + k^2 + \ell^2)^2} \quad . \tag{4.6}$$

As pointed out by Neerfield[88] and Hill[89] values of S_1 and $S_2/2$ usually lie between the values calculated by the limiting assumptions of Voight and Ruess. Therefore, arithmetic mean values of the elastic constants calculated from the two models are used in the computer program. Input of the three single crystal constants, S_{11}, S_{12}, and S_{44} are requested in lines 24-26 of Fig. 4.4. The calculated value of $1+\nu/E$ is typed and used to determine the stress.

4.3.10 Calculation of Instrumental Error

The instrumental error is determined if requested. The necessary information input in lines 30-33 of Fig. 4.4 includes the divergent slit, the sample displacement, the ψ-axis missetting and the goniometer radius. If these parameters are not input, the instrumental error is not printed in the output, as in Fig. 4.6. The exact aberration (and its sign) due to horizantal beam divergence, sample displacement, and ψ-axis displacement, are determined <u>after</u> all peak positions have been determined.

In the initial dialog, the operator may desire a preset total error in stress. From this, the statistical counting error must be obtained using Eq. 4.1. Therefore, an estimate of the instrumental error is made by using the approximate peak location input by the operator at line 6, Fig. 4.5. This gives a reasonable estimate of the instrumental error before the actual peak angles are determined in order to calculate the desired counting error.

4.3.11 Position Sensitive Detector Program

The use of a position sensitive detector (PSD) for residual stress measurements has been treated previously. A complete description of

the detector system and performance was presented in ONR TR Nos. 11, 12 and 15. The PSD eliminates all 2θ motion and enables data over the entire diffraction profile to be accumulated simultaneously. The data is stored in a multichannel analyzer (MCA).

The automated residual stress package allows the data to be transferred from the MCA to the computer for processing. The program features are described below and assume the reader has sufficient knowledge of operation of the PSD system. Also, previous calibration of the detector is necessary.

Figure 4.9 gives a typical dialogue for the position sensitive detector program. The two tilt, $\sin^2\psi$ or Marion-Cohen methods may be used with the same features as explained in Sec. 4.3.2. In order to transfer the data from the MCA to the computer, the first channel and last channel on the MCA to be transferred must be input (lines 2-3, Fig. 4.9).

Information to determine the calibration of position along the detector to °2θ must be input in lines 4-6 of Fig. 4.9. This data must be determined from a calibration sample.
The linear calibration constant in °2θ/channel on the MCA is input in line 4. The absolute reference angle in °2θ is input with the corresponding channel on the MCA.

The Lorentz-polarization, absorption and scattering factor corrections are made to the data after the angular position of each data point is determined by the computer program. The program next determines the region of parabolic fit by including all data points within 85 pct of

USING POSITION SENSITIVE DETECTOR

```
   -METHOD OF RESIDUAL STRESS ANALYSIS-
 1 TWO TILT METHOD  <Y/N>        Y

 2 FIRST CHANNEL #               170
 3 LAST  CHANNEL #               204

   -CALIBRATION DATA-
 4 DEGREES 2THETA/CHANNEL #      .04
 5 2THETA          156.14
 6 CHANNEL #       191

   -INITIAL INFORMATION-
 7 CURRENT PSI                   0
 8 TIME  (SEC)                   10

   -PSI TILTS-
 9 PSI(  1 )  0
10 PSI(  2 )  45

   -STRESS CONSTANT DATA-
11 STRESS CONSTANT 'K'  <Y/N> N
12 WAVELENGTH             2.2909
13 SCATTERING FACTOR CORR. ? <Y/N> N
14 EXPT. ELASTIC CONSTANTS <Y/N> Y
   (1+U)/E                .43E-7

15 INSTRUMENTAL ERROR ANALYSIS <Y/N> N

16 HOW MANY REPEATED MEASUREMENTS  1

   TYPE ALT MODE TO CONTINUE

 PSI =    0.00                    ENABLE 'START READOUT' OF MCA

  CH. NO.     <---              OBSERVED CPS              --->

    154        128.60   141.60   148.00   163.10   159.60
    159        166.90   160.80   163.90   163.80   145.60
    194        147.60   143.80   133.30   131.90

 2THETA       <---       .      CORRECTED CPS             --->

  155.900       67.29    73.86    77.19    85.06    83.22
  156.100       87.01    83.82    85.43    85.36    75.87
  156.300       76.90    74.91    69.43    68.69


 PEAK             156.145
 ERROR              0.0063275

 PSI =   45.00                    ENABLE 'START READOUT' OF MCA

  CH. NO.     <---              OBSERVED CPS              --->

    182        120.60   124.80   142.70   139.40   154.70
    187        153.50   157.30   158.40   157.60   154.20
    192        155.10   145.80   142.80   132.20   128.40


 2THETA       <---              CORRECTED CPS             --->

  155.820       80.09    82.83    93.33    92.41   102.49
  156.020      121.63   124.89   124.75   124.16   121.72
  156.220      102.39    96.19    94.16    87.11    84.49


 PEAK             156.112
 ERROR              0.0061249


 SIN**2 PSI        D(I)          SIGMA (I)
    0.000        1.172727        0.0002193
    0.500        1.172799        0.0002187

 STRESS                        3326 PSI
 STATISTICAL ERROR     +/-     1869 PSI
```


FIGURE 4.9 Input/output from PSD version of STRESS.

the maximum intensity. A least-squares parabolic fit is then made utilizing all the data points within the region. Statistical counting errors are determined for the peak location.

The number of ψ tilts, elastic constant data and instrumental error data are similar in form to the program section for the diffractometer. All calculations for the stress, counting errors and instrumental errors are identical to those previously described in the diffractometer version of the residual stress package.

Due to the physical size of the PSD, it is not possible to place the detector on the focusing circle in back reflection geometry. This is clearly seen in Fig. 4.10 where the perpendicular bisector of the detector anode is placed at 156°2θ, a typical back reflection angle for use with steel specimens. The length of the anode and the surrounding packaging require the detector to be placed behind the X-ray tube and off the focusing circle by 5 cm.

For the portable residual stress analyzer to be described in ONR TR No. 18 this defocussing is minimized because of the size of the X-ray tube.

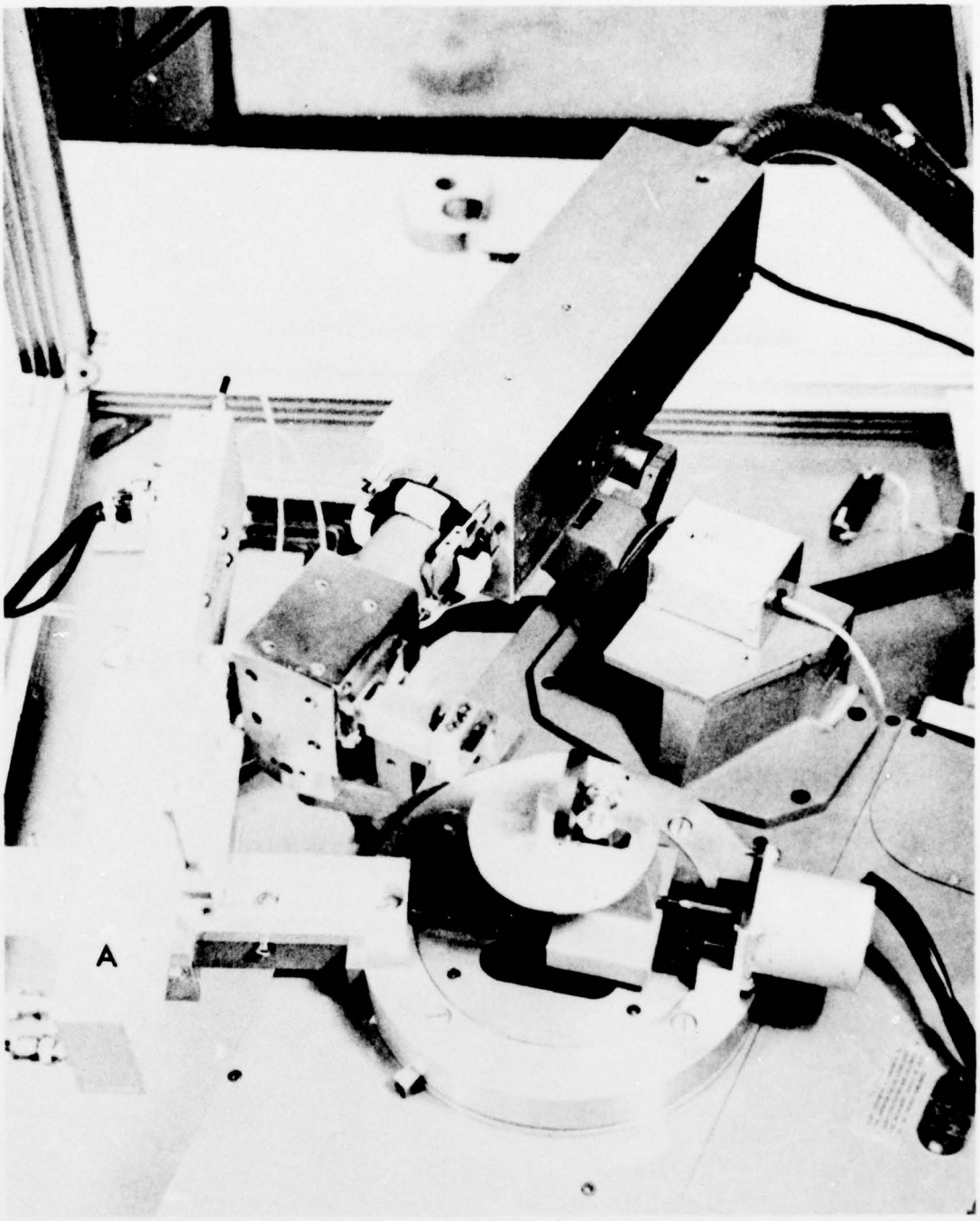

FIGURE 4.10 Top view of Picker diffractometer showing the location of the position sensitive detector (A). Due to the physical size of the detector, it was placed 5 cm from the focusing circle at $\psi=0^{\circ}$.

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13. ABSTRACT

The software design of a complete computer controlled X-ray residual stress is described. The program will operate using either a position sensitive detector or a diffractometer for data accumulation.

For use with a diffractometer, optimization of the time of data accumulation for a prescribed statistical counting error is presented. The peak location routine using a multiple pass refinement of the peak position is detailed.

The software allows operator intervention during operation and specification of either the 'two tilt' or '$\sin^2\psi$' technique. The Marion-Cohen technique is automatically accomplished if oscillations in d vs. $\sin^2\psi$ are evident. Provisions for the receiving slit movement, counting statistical analysis and instrumental error analysis are provided. The operator specifies the total error in stress he will tolerate, and the program then optimizes the counting strategy.

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